

## From the Researcher's Notebook

### Catalytic Transformations of Fluids

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**Abstract**—Fluids play a very important role in processes of the chemical and physical evolution of crustal and mantle rocks. The influence of certain rocks on chemical reactions between fluid components is studied. Experimental results of the catalytic activity of serpentinite, ophitic gabbro, and lizardite–antigorite asbestos relative to vapor conversion of methane (CH<sub>4</sub>) have been obtained with the formation of syngas at atmospheric pressure and temperatures up to 850°C. Such transformations are assumed to be able to take place under natural conditions in fluid systems that accompany massifs of seabed ultrabasic rocks, as well as kimberlite pipes. Proceeding from the studies performed, options of the practical use of various crustal rocks as catalysts of fluid transformations have been proposed. The experimental results confirm the initial theses of the authors' concept with respect to the role of heterogeneous–catalytic mechanisms of transformations of fluids in the Earth's crust.

**Keywords:** fluids, catalytic influence of crustal rocks and minerals, H<sub>2</sub> and CH<sub>4</sub> emission, serpentinite, lizardite–antigorite asbestos, ophitic gabbro, catalytic activity, use of the results obtained in real technological processes.

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Fluids—highly mobile supercritical mixtures of H<sub>2</sub>O, CO<sub>2</sub>, CH<sub>4</sub>, CO, H<sub>2</sub>, and other components—play the leading role in processes of the physicochemical evolution of crustal and mantle rocks. The presence of a fluid phase accelerates reactions between minerals and the growth and dissolution of mineral grains by several orders of magnitude and intensifies melting processes and polymorph transformations, which, in turn, influences the development of various types of deformations in mineral aggregates. Obviously, the opposite effect of rocks and minerals on equilibrium in fluid systems themselves, as well as on their component composition, is also possible. Along with geochemically typical processes, such as reduction–oxidation, dehydration–hydration, and decar-

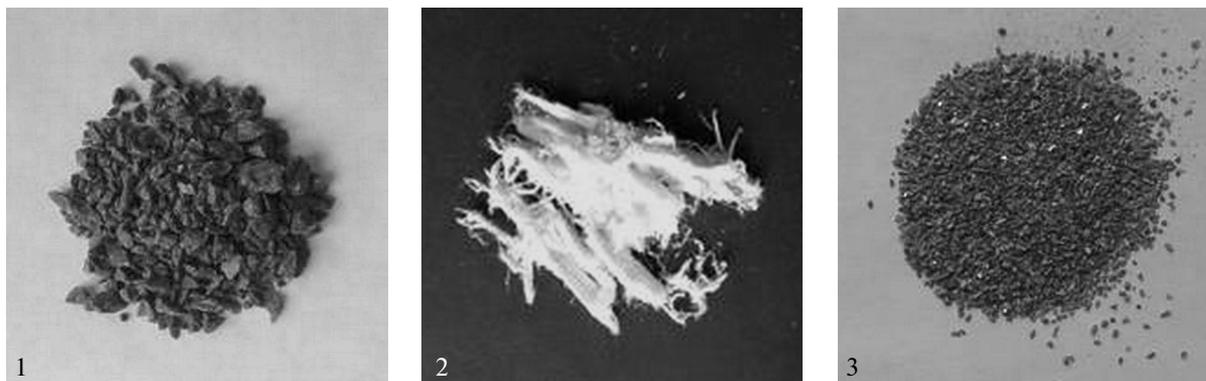
bonation–carbonation reactions, which are controlled by mineral associations of rocks, there exist processes of other types, which can be characterized as heterogeneous–catalytic reactions. They either do not change or change insignificantly the composition of rocks themselves but affect the character and rate of reactions between components of fluids.

The catalytic influence of rocks and minerals on chemical reactions between the components of fluids is a poorly studied topic in the geochemistry of fluid–mineral interactions. Geochemical publications actively discuss only the possibilities of catalytic reactions of the synthesis of hydrocarbons in natural processes by the Fischer–Tropsch mechanism as a result of hydrogen interaction with CO<sub>2</sub> and CO in the presence of metal-bearing catalysts:



The attention of geochemists to reactions of this type is explained by finds of diverse hydrocarbons in fluid inclusions in minerals of magmatic and metamorphic rocks. Such mechanisms are drawn for explanation of the abiogenic origin of hydrocarbons at late stages of the evolution of CO<sub>2</sub>- and CO-bearing fluids that accompany nepheline syenite and alkaline granite complexes [1–3]. In this case, postmagmatic reactions of replacement (for example, alkaline amphiboles

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**Fig. 1.** Serpentinite sample from the Bogorodskoe deposit (South Urals). Basic oxides in the chemical composition of the rock after crushing (wt %).

**Fig. 2.** Lizardite–antigorite asbestos sample. Basic oxides in the chemical composition of the rock (wt %).

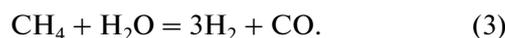
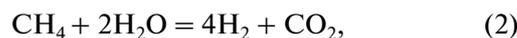
**Fig. 3.** Ophitic gabbro sample. Basic oxides in the chemical composition of the rock after crushing (wt %).

with aegirine) are considered as a source of hydrogen and magnetite ( $\text{Fe}_3\text{O}_4$ ) occurring in rocks as the main catalyst. In turn, the catalytic Fischer–Tropsch reactions are considered as the main source of the formation of methane and more complex hydrocarbons in hydrothermal transformation processes, particularly during serpentinization, i.e., the transformation of ultrabasic and basic compositions into serpentinite [4–7]. The catalysts of these reactions could be awaruite ( $\text{FeNi}_3$ ) and chromite ( $\text{FeCr}_2\text{O}_4$ ), which are usually present in serpentinites [8].

However, in natural conditions, other transformations, catalyzed by rocks or individual minerals, are also possible. Moreover, the analysis of routes of catalytic transformations of fluids in the Earth's crust demonstrates the geological reality of processes analogous to industrial technologies. Indeed, the chemical composition and structures of many rocks are identical to classical synthetic catalysts and are metal-doped silicate and alumina matrices. Figuratively speaking, the Earth's crust in terms of its catalytic potential should be attributed to giant catalytic objects. Even facts of changes in the catalytic properties of rocks as a result of their interaction with fluid flows [9, 10] by no means discredit the proposed catalytic concept of the transformation of fluids in the Earth's crust. Similar phenomena accompany processes with the participation of synthetic catalytic materials that often require introducing the classical additional operation of catalytic matrix regeneration.

Along with the Fischer–Tropsch mechanism-based synthesis of hydrocarbons and their oxygen-bearing derivatives, the heterogeneous–catalytic processes that can occur in natural conditions include, for example, the catalytic synthesis of ammonia from  $\text{H}_2$  and  $\text{N}_2$  (the Haber synthesis) or reactions of transformations of hydrocarbons with the formation of solid carbon. In particular, we cannot exclude that a reac-

tion accompanied by the formation of hydrogen, i.e., the reaction of  $\text{CH}_4$  vapor conversion, known in industrial practice as synthetic gas production, can really proceed in terrestrial conditions on natural catalysts:



Note that  $\text{CH}_4$  and  $\text{H}_2\text{O}$  mixtures are among the most abundant fluids in the Earth's lithosphere. To substantiate the hypothesis proposed, we performed experimental studies of the catalytic activity of crustal rocks in the reaction of methane vapor conversion [9, 10].

The following rocks were chosen for the experiments:

- *massive serpentinite* from the Bogorodskoe deposit (South Urals) (Fig. 1), which is composed of two serpentine polymorphs, namely antigorite and lizardite. It also contains accessory chromite ( $\text{Mg, Fe}(\text{Cr, Al})_2\text{O}_4$ ) with magnetite rims and rare secondary chlorite. The rock has a fine-pored fiber texture with massive aggregates of lizardite and striplike manifestations of antigorite;

- *lizardite–antigorite asbestos* (Bazhenovskoe deposit, South Urals) (Fig. 2), accessory minerals being represented by a small quantity of magnetite; and

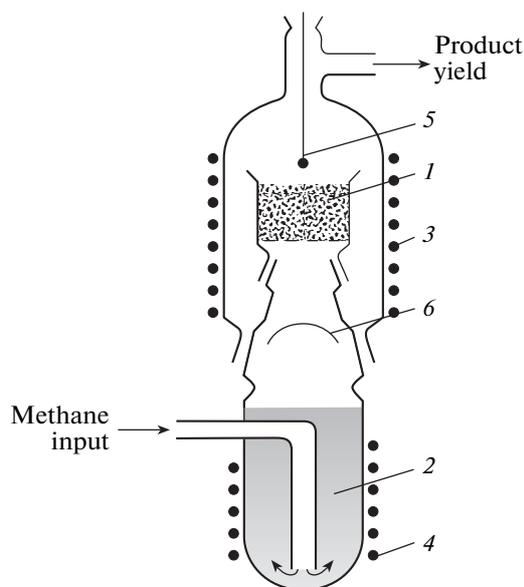
- *ophitic gabbro* dredged from the hydrothermal Logachev Field on the Mid-Atlantic Ridge (Fig. 3). The rock has a coarse texture determined by crystals of strongly altered clinopyroxene and plagioclase. Clinopyroxene (augite) is replaced by secondary amphibole (actinolite or Al-bearing actinolite) and chlorite. Plagioclase (labradorite–bytownite) is sericitized and occasionally replaced by zoisite. Gabbro contains

magnetite  $\text{Fe}_3\text{O}_4$ , titanomagnetite  $\text{Fe}(\text{Fe}, \text{Ti})_2\text{O}_4$ , ilmenite ( $\text{FeTiO}_3$ ), and pyrite ( $\text{FeS}_2$ ).

In terms of the composition ( $\text{MgO-SiO}_2$  base doped by catalytically active components Fe, Ni, Cr) and texture (fine-fibrous and fine-porous matrix), serpentinite and asbestos are quite similar to artificial catalytic materials traditionally applied in industrial catalysis.

Note that the samples were only crushed and did not undergo any further chemical or mechanical treatment. A particle-size crushed-rock fraction of 0.5–0.7 mm was used in the experiments, which were performed using a flow-type quartz glass catalytic reactor (Fig. 4). The volume of the serpentinite catalyst charged into the reactor tank was  $5.3 \text{ cm}^3$ , the height of the catalytic layer being 1 cm. The methane flow rate ( $20 \text{ cm}^3/\text{min}$ ) was regulated by an automatic flowmeter; the water content in the flow was maintained by changing the temperature in the bubbling element (this parameter was calibrated by the fall of water from the bubbling element). The vapor/methane volume ratio at the entry to the reaction zone ranged from 8/1 to 10/1; the linear filtration rate of the reaction stream through the catalytic layer was 0.5–0.6 cm/s; and the time of the contact of the reaction stream with the catalytic layer was 1.5–2.0 s. The experiments were performed at ambient pressure and 500–850°C. Heating and temperature regulation in the catalysis zone were performed using an electric heater with a thermocouple control. The content of components at the inlet and that of transformation products at the outlet were determined using a Crystal-5000 chromatograph. The assessments performed on the basis of the experimental results obtained at  $\sim 800^\circ\text{C}$  are determined by methane conversion to hydrogen at a level of 2.4 mL from 1 g of serpentinite over 20–30 min. These assessments constituted the foundation for developing catalytic reactors to produce superpure hydrogen based on vapor conversion of methane with the use of membrane blocks of selective hydrogen release, which was proposed by the authors of this article.

The initial samples and run products were investigated on CamScan MV2300 (VEGA TS 5130MM) electron microscopes with an energy-dispersive microanalyzer INCA-Energy-250 at the RAS Institute of Experimental Mineralogy and Jeol JSM-6480LV electron microscopes with an INCA-Energy-350 energy-dispersive microanalyzer and an INCA-Wave-500 wavelength microanalyzer in the Department of Petrology, Moscow State University. The Raman spectra of samples before and after the experiments were obtained on a Raman JY Horiba XPLoRa Jobin spectrometer in the same department using a 532-nm laser. The phases on Raman spectra were identified using the database <http://rruff.geo.arizona.edu/rruff/>. Powder X-ray diffraction patterns of run products were obtained on a PC/HZG-4 auto-



**Fig. 4.** Reactor configuration for using vapor conversion of methane on serpentinite. (1) A tank with serpentinite granules; (2) a gas bubbler with water; (3, 4) electric heating elements for the reactor zone and gas bubbler; (5) thermocouple; (6) a spray trap.

matic diffractometer at the RAS Institute of Experimental Mineralogy.

The experiments revealed that the conversion of  $\text{CH}_4$  to  $\text{H}_2$  on serpentinite increases with temperature and reaches 14% at  $825^\circ\text{C}$  (table). In the absence of the catalyst (serpentinite) at the temperatures specified in the table, the reaction does not proceed and hydrogen is not formed, which convincingly proves the catalytic influence of natural serpentinite. The conversion of methane to  $\text{CO}$  and  $\text{CO}_2$ , which is 3% for both components, is observable only under catalysis. An unexpected result was that the transformation products included the alcohols  $\text{CH}_3\text{OH}$  and  $\text{C}_2\text{H}_5\text{OH}$ , which do not form on standard catalysts in the technological process of vapor conversion of methane. The presence of more complex oxygen-bearing hydrocarbons in the run products cannot be excluded either.

The experiments conducted make it possible to state that serpentinite has satisfactory catalytic properties relative to the methane vapor conversion process. Under a very short contact of the fluid water–methane flow with this rock, sufficiently high degrees of the conversion of  $\text{CH}_4$  to hydrogen- and oxygen-bearing compounds were registered. Therefore, serpentinite can be considered as an effective natural catalyst.

The X-ray powder diffraction of the serpentinite sample after the experiment showed the presence of forsterite ( $\text{Mg}, \text{Fe})_2\text{SiO}_4$  in it, which was also confirmed by Raman spectra; the latter also fixed the pres-

Results of the chromatographic analysis of the investigated samples of natural catalysts, vol %

Temperatur, °C	Serpentinite		Asbestos		Gabbro	
	H <sub>2</sub>	MC, %	H <sub>2</sub>	MC, %	H <sub>2</sub>	MC, %
600	traces	0	1.0 (0.1)	4.0 (5.0)	0.4	4.0
700	2.0	6.0	1.7 (0.4)	4.0 (3.0)	1.1 (0.2)	5.0 (2.5)
800	n.d.	n.d.	4.7 (2.4)	7.2 (5.0)	1.8	6
810	7	13.0	n.d.	n.d.	n.d.	n.d.
825	n.d.	n.d.	7.0 (2.4)	10.0 (9.0)	n.d.	n.d.
845	9.3	14	n.d.	n.d.	4.0 (2.3)	9.0 (6.0)

MC is methane conversion; in parentheses, measurement results under reheating are given; n.d. means not determined at a given temperature

ence of a small amount of undecomposed antigorite. The structure of the sample after the experiment remained practically unchanged. However, as opposed to the initial serpentinite, where the number of oxides in microprobe analyses does not exceed 80 wt %, the number of oxides in the samples of microprobe analyses of the run products varies from 87 to 99 wt % (see table). This testifies to the very high degree of dehydration of the sample. To all appearances, the process of the interaction of the rock with the fluid H<sub>2</sub>O + CH<sub>4</sub> is accompanied by a small yield of SiO<sub>2</sub>; as for the matrix, after the experiment, it was most likely an X-ray amorphous substance compositionally close to serpentine–anhydrite (Mg, Fe)<sub>3</sub>Si<sub>2</sub>O<sub>7</sub>.

Thus, the process of the dehydration of serpentinite in the presence of the fluid H<sub>2</sub>O + CH<sub>4</sub> in the temperature interval from 500 to 825°C is close to that of the “dry” dehydration of serpentinite under heating, the initial stages of which are characterized by the active crystallization of forsterite and the formation of X-ray amorphous anhydrite. The after-experiment sample lacked chromite, the latter being fully replaced by magnetite.

Similar transformations were observed in asbestos as well. According to X-ray phase analysis data, in the course of the experiments, lizardite–antigorite asbestos, which resembles serpentinite in terms of composition and structure, transformed into forsterite olivine when interacting with CH<sub>4</sub> + H<sub>2</sub>O fluids in the temperature interval from 600 to 825°C. At the same time, after the experiments, the total number of oxides in samples of the specimens studied varied from 90 to 99 wt % as opposed to 70–78 wt % for the initial asbestos, which testifies to the fact that dehydration processes proceed in the presence of CH<sub>4</sub> + H<sub>2</sub>O fluids. The run products also contained a small amount of magnetite. The hydrogen yield increased from 1 vol % at 600°C to 7 vol % at 825°C (see table). The level of the conversion of methane to H<sub>2</sub> in the presence of asbestos was considerably lower than in the presence of serpentinite. Nevertheless, it also increased with the growth of the temperature from 4 vol % at 600–700°C

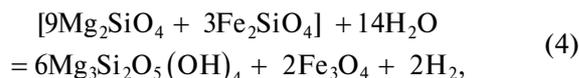
to 10 vol % at 825°C. The gas-phase products showed only traces of CO and CO<sub>2</sub>. When reheated, asbestos samples sharply became less catalytically active: the hydrogen yield was only 0.1–2.4 vol %, while the level of CH<sub>4</sub> conversion was between 5 and 9 vol % in the same temperature interval. As opposed to experiments with the use of serpentinite, the run products for asbestos samples contained no traces of any oxygen-bearing hydrocarbons.

As for ophitic gabbro samples, their catalytic activity turned out to be minimal. The hydrogen yield increased from 0.4 vol % at 600°C to 4 vol % at 845°C (table). The level of the conversion of methane to H<sub>2</sub> in the presence of gabbro also increased with the growth of the temperature from 4–5 vol % at 600–700°C to 9 vol % at 845°C. The gas-phase products contained only traces of CO and CO<sub>2</sub>. Just like in the case with asbestos, the reheating of gabbro samples led to a sharp decrease in their catalytic activity: the hydrogen yield was 2.4 vol %, and the maximal level of CH<sub>4</sub> conversion was about 6 vol % within the same temperature interval.

The high effectiveness of serpentinite as a catalytic material relative to vapor conversion of methane makes it possible to hypothesize on the role of this process in some geochemical and geological processes that can involve H<sub>2</sub>O + CH<sub>4</sub> fluids. However, the experiments conducted allow us to judge only about near-surface processes thus far because the influence of pressure on the reactions under consideration requires additional studies.

The process of serpentinitization requires close attention first and foremost. As is known, serpentinites are powerful sources of H<sub>2</sub> and CH<sub>4</sub> [5], the anomalies of which are registered in seawater columns above the massifs of ultrabasic rocks of the sea floor and those of ophiolites [4, 6]. The appearance of serpentinite is tra-

ditionally interpreted as a result of serpentinization of ultrabasic rocks according to the exothermal reaction:



i.e., olivine + H<sub>2</sub>O = serpentine + magnetite + H<sub>2</sub>. As for the appearance of CH<sub>4</sub>, it is usually related to catalytic processes of the Fischer–Tropsch reaction type (1) at relatively low temperatures [5, 6]. Awaruite (FeNi<sub>3</sub>), magnetite, and chromite (FeCr<sub>2</sub>O<sub>4</sub>) usually occurring in serpentinite are considered as catalysts of these reactions during serpentinization. The experiments conducted show that serpentinization of ultrabasic rocks (4) is not the only mechanism of the formation of H<sub>2</sub> associated with serpentinite massifs on the sea floor. This component can also be generated at the expense of catalytic reactions of types (2) and (3) in processes connected with the filtering of deep water–methane fluids through the formed massifs of serpentinites. However, according to the experimental results, the temperatures at which the vapor conversion of methane takes place (more than 800°C) are much higher than the temperatures of hydrothermal systems (150–450°C) that transform seabed ultrabasic rocks. Indeed, we know massifs of seabed peridotites, the hydrothermal alteration temperatures of which reach 900°C. Hence, we should not exclude the possibility that the catalytic production of H<sub>2</sub> can take place in such massifs. It is also possible in areas of active volcanism, when significant masses of hot basalt magma come close to the surface. The processes described by reactions (2) and (3) reflect the high-temperature stages of catalytic transformations of fluid flows. If the temperature decreases, they can be replaced by other processes, for example, by Fischer–Tropsch reactions, which use hydrogen produced at higher temperatures.

We can assume that catalytic transformations of fluids (2) and (3) take place, in particular, in the course of the evolution of the fluid phase of kimberlite pipes. Serpentine associated with magnetite, chromite, and other catalytically active minerals [8] is one of the main components of most kimberlites, and the temperatures of the eruption of kimberlite magma near the surface can quite easily reach 800–900°C. Note that intensive flows of H<sub>2</sub> and CH<sub>4</sub> (up to 105 m<sup>3</sup>/day), as well as the presence of hydrocarbons, were really registered in the boreholes of some kimberlite pipes of Yakutia, for example, Udachnaya Pipe. We cannot exclude that H<sub>2</sub> in such flows acts as a product of vapor conversion of CH<sub>4</sub> in the presence of the water component of fluids that accompany kimberlite magmas. As we see it, such volumes could allow for organizing the conversion of methane directly in places of its emissions using relatively simple hardware equipment.

In our opinion, the results of studying this process can also help develop ideas on the mechanisms of

ozone layer degradation in the atmosphere. Until recently, it was believed that the main cause of ozone layer destruction was anthropogenic emissions of Freon aerosols into the atmosphere. However, not long ago, work [11] proposed an alternative version, which assigns the main role in the destruction of ozone to hydrogen that ascends from the subsoil. In this respect, catalytic transformations of fluids on crustal rocks should be considered as quite a real and debatable mechanism of the formation of extraneous hydrogen and its emission from the Earth's crust into the atmosphere [9, 10].

The experimental data obtained prove the unquestionable prospects of the use of natural catalysts in industrial technological processes. One can also say with reason that the hypothesis about the emission of hydrogen from the Earth's crust into the atmosphere in the course of catalytic reactions of fluids in the presence of crustal rocks deserves closer attention at least as a matter of scientific discussion.

The results of our experiments testify to the necessity to organize systemic studies of catalytic properties of a wide range of crustal rocks relative to the diverse routes of chemical transformations of fluid components, as well as to the unquestionable prospects of the currently poorly studied trend, catalytic geochemistry. Obviously, in solving these tasks, we should abandon the classical geochemical approach based on thermodynamic equilibrium and take into consideration the fact that chemical processes in the Earth's crust proceed in a nonequilibrium regime.

Further development of the catalytic concept in geochemical science can lead to significant practical results. It is possible that, in the course of studies of catalytic properties of various crustal rocks, scientists will establish a reason for industrial excavation of the most catalytically active rocks that are considered as an economically effective alternative to expensive synthetic catalytic materials. Such rocks will find application as, for example, a catalytic block introduced directly in the passage for engine fuel. This approach is applicable in solving the important practical task associated with transferring engine building to hydrogen-containing fuels to increase the efficiency of engines and improve their environmental performance [12]. Hydrogen-containing natural emissions can also be used to obtain cheap superpure hydrogen, which is in high demand in various industrial processes. As was mentioned, to solve this task, we have developed a palladium hydrogen-filtering membrane block.

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SPELL: 1. aegirine, 2. undecomposed